

440 Rec'd PCT/PTO 11 SEP 2000

FORM PTO 1390
(REV 5-93)

US DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY DOCKET NO.
2000_1212A

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 USC 371**

U.S. APPLICATION NO.
(if known, see 37 CFR 1.55)
NEW **09/623975**

International Application No. **PCT/NO99/00008**

International Filing Date
January 11, 1999

Priority Date Claimed
March 13, 1998

Title of Invention
CATALYST FOR THE SYNTHESIS OF AMMONIA FROM HYDROGEN AND NITROGEN

Applicant(s) For DO/EO/US
Terje FUGLERUD and Per Torbjorn SKAUGSET

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 USC 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 USC 371.
3. ☐ This is an express request to begin national examination procedures (35 USC 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 USC 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 USC 371(c)(2))
 - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau). **ATTACHMENT A**
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 USC 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 USC 371(c)(3)).
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 USC 371(c)(3)).
9. ☒ An executed oath or declaration of the inventor(s) (35 USC 371(c)(4)). **ATTACHMENT B**
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 USC 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98. **ATTACHMENT C**
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. **ATTACHMENT D**
13. ☐ A **FIRST** preliminary amendment.
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

**THE COMMISSIONER IS AUTHORIZED
TO CHARGE ANY DEFICIENCY IN THE
FEE FOR THIS PAPER TO DEPOSIT
ACCOUNT NO. 23-0975.**

430 Rec'd PCT/PTO 1.1 SEP 2000

U.S. APPLICATION NO. (if more than one, see 37 CFR 1.53) NEW 09/623975		INTERNATIONAL APPLICATION NO. PCT/NO99/00008		ATTORNEY DOCKET NO. 2000 1212A			
17. [X] The following fees are submitted BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)): <input checked="" type="checkbox"/> Search Report has been prepared by the EPO or JPO \$840.00 <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) \$670.00 <input type="checkbox"/> No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) \$690.00 <input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$970.00 <input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-33(4) \$ 96.00 ENTER APPROPRIATE BASIC FEE AMOUNT =				CALCULATIONS		PTO USE ONLY	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$			
Claims	Number Filed	Number Extra	Rate				
Total Claims	- 20 =		X \$18.00	\$			
Independent Claims	- 3 =		X \$78.00	\$			
Multiple dependent claim(s) (if applicable)			+ \$260.00	\$			
TOTAL OF ABOVE CALCULATIONS =				\$840.00			
Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28)				\$			
SUBTOTAL =				\$840.00			
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				+		\$	
TOTAL NATIONAL FEE =				\$840.00			
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (\$40 per property).				+		\$40.00	
TOTAL FEES ENCLOSED =				\$880.00			
				Amount to be refunded:		\$	
				charged:		\$	

09/623975

422 7000 PCT/PIO 11 SEP 2000

- a. ☒ A check in the amount of \$880.00 to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. 23-0975 in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 23-0975. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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September 11, 2000
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2000_1212A

CATALYST FOR THE SYNTHESIS OF AMMONIA FROM HYDROGEN AND NITROGEN

The present invention relates to a catalyst for the synthesis of ammonia from hydrogen and nitrogen.

The ammonia synthesis catalyst play an important role in the manufacture of ammonia. It effects both the economics and operating conditions of ammonia plants.

Industrial catalysts for ammonia synthesis must satisfy a number of requirements: high catalyst activity at the lowest possible reaction temperatures in order to take advantage of the favourable thermodynamic equilibrium at low temperatures, good resistance to poisoning by oxygen-, chlorine- and sulphur -containing compounds, long life and high mechanical strength.

In order to save energy the synthesis pressure has been steadily reduced from 250-350 bar to 80-150 bar during the last 30 years due to optimised operation of the synthesis loop made possible by new technology, especially for fabrication of the converter. Improvements in the front end has drastically reduced the content of catalyst poisons (oxygen-, sulphur- and chlorine-containing compounds) in the gas inlet to the converter. However, the drop in synthesis pressure necessitates a three- to four-fold increase in the catalyst volume. The demands upon the catalyst quality has also increased. Apparently small improvements in the catalyst activity may lead to major improvements of the operation of a modern ammonia synthesis loop.

The ammonia synthesis catalyst precursor, the unreduced catalyst, is manufactured by melting iron oxides, mainly magnetite, and oxides or carbonates of Al, K, Ca and Mg, the so-called promoters. The melting is performed in electrical furnaces at a temperature of above 1600 °C. The ratio $\text{Fe}^{2+}/\text{Fe}^{3+}$ in the melt is normally in the range 0.5-0.75. The melt is then poured into metal trays where it solidifies and cools. The

solid precursor is then broken up, crushed and sieved to obtain the required grain size.

The precursor is reduced to active iron catalyst "in situ" in the ammonia synthesis converter or used to manufacture the prereduced catalyst by a complete reduction under optimised conditions in a prereduction reactor. This material is pyroforic, but after a skin oxidation of the active surface with oxygen, it can be handled safely in air at ambient temperatures.

The promoters mentioned above, containing Al, K, Ca and Mg, are essential for the formation of a large surface of iron in the reduced catalyst and for the kinetics of ammonia formation.

Since the development of the catalyst started, about 90 years ago, the concentration of these promoters have been optimised to obtain maximum activity and to meet the additional requirement mentioned above. Further improvements seem only possible by adding new promoters, or new combination of promoters.

Cobalt oxide has been used as such a new promoter.

From US patent 3839229 it is known an ammonia synthesis catalyst where cobalt oxide is used as a promoter. The catalyst consists essentially of a solid solution of iron oxide and cobalt oxide and said cobalt oxide is present in an amount of 5 to 10 percent by weight expressed as cobalt. In the catalyst there is further included a promoter selected from the group consisting of alumina, silica, zirconia, magnesia, lime (CaO), potassium oxide and rare earth metal oxides.

Another possible promoter is titanium oxide. Only few catalysts using this promoter have been previously described. S.A. Abdukadyrova et al. have in "Tr.Mosk.Khim.-Tekhnol.Inst. (1970), No.2, 122-5", described that titanium oxide improves the thermal resistance but reduces the activity.

Titanium oxide used as a structural promoter is described by M. E. Dry et al in "Journal of Catalysis", 6, page:194-199, 1966. Titanium oxide is not so effective as aluminium oxide with regard to surface area. This area and the volume of

chemisorbed CO normally increase with promoter content, but in contrast to the other promoters (Al_2O_3 , MgO , CaO) TiO_2 had a maximum in the volume of chemisorbed CO at about 0.5 g atom cation per 100 g atom Fe.

The main object of the present invention was to develop a catalyst for ammonia synthesis with improved activity.

Higher activity could be utilized in two ways: High reaction rate at high ammonia concentrations or higher activity at low temperatures.

The catalyst activity may be expressed as a rate constant in a rate equation for the synthesis reaction. The rate is a function of temperature, pressure and the composition of the gas, and decreases rapidly with increasing ammonia concentrations. Thus a large fraction of the catalyst volume in a synthesis converter will have ammonia concentration approaching the exit concentration. Hence, to improve the efficiency, increasing the reaction rate at high ammonia concentration is of particular interest.

As the equilibrium temperature decreases with increasing ammonia concentration, searching higher conversion by increasing the reaction rate at high ammonia concentration also means searching a catalyst with higher activity at low temperature.

The inventors have manufactured a lot of samples of iron oxide based catalysts with various promoters in various concentrations to improve the catalyst activity. In addition to the conventional promoters mentioned above, new promoters have been tested, in particular cobalt- and titanium oxide.

The inventors found that the reaction rate at high ammonia concentration increased with 10-20% when both cobalt- and titanium oxide were used as promoters together with the conventional promoters.

The most preferred catalyst was achieved when the concentration of cobalt was between 0.1% and 3.0 % by weight of metal and when the concentration of titanium was between 0.1 % and 1.0 % by weight of metal.

The atomic ratio $\text{Fe}^{2+}/\text{Fe}^{3+}$ was between 0.5-0.65.

The present invention will thus in its widest scope comprise a catalyst for the synthesis of ammonia from hydrogen and nitrogen consisting of iron oxides and promoters where the promoters comprise oxides of both cobalt and titanium in addition to Al, K, Ca and Mg oxides.

The invention will be further explained in the example.

Example:

Samples were made by mixing iron ore, mainly magnetite, Fe_3O_4 , with promoters. These mixtures were then melted in a ceramic crucible in a laboratory furnace. The temperature was kept at about 1600 °C. The ratio of $\text{Fe}^{2+}/\text{Fe}^{3+}$ was adjusted to within 0.5 to 0.65 by adding Fe-metal to the melt.

The melt was then poured into an iron crucible and cooled.

The concentration of the promoters in the samples varied as shown in table I:

Table I:

K	0.4 - 0.5
Ca	1.6 - 1.8
Mg	0.3 - 0.5
Al	1.5 - 1.8
Co	0.10- 3.00
Ti	0.14- 0.95

The balance being iron oxides with natural impurities. The cooled samples were crushed and sieved to 0.4 - 0.63 mm particles for testing in a microreactor.

The samples, (10 g each), were tested together with the reference sample in a microreactor. The reference sample was a conventional catalyst with no cobalt- or titanium oxide added.

The samples were reduced in a flow of H_2 and N_2 in the ratio 3/1 at a space velocity of 33000 1/h. The increase in temperature was $3^\circ C/h$ from $250^\circ C$ to $520^\circ C$ and $520^\circ C$ was kept for 24 hours to ensure complete reduction.

The samples were then tested at a pressure of 50 bar, space velocity in the range 8000 to 50000 1/h, and temperatures in the range $350^\circ C$ to $420^\circ C$. The inlet gas was $H_2/N_2=3/1$ with an ammonia concentration of 1.0 to 1.2 % (vol.). Exit ammonia concentration was in the range 4 to 9 % (vol.).

The data obtained during a period of about 100 hours and after an initial stabilisation of 24 hours or more, were used to calculate parameters in a kinetic model for the reaction. The model was then further used for calculating the relative activity and relative reaction rate at higher ammonia concentration than used in the test.

The following table II shows the concentration of cobalt and titanium in the various samples, average relative catalyst activities and predicted relative reaction rate at higher ammonia concentration (20% NH_3 , $420^\circ C$) calculated by the kinetic model.

Table II:

Sample	Co w %	Ti w %	Average Relative Catalyst Activity at 350°C	Relative Reaction Rate (20% NH ₃ and 420°C)	Average Relative Catalyst Activity at 420°C
1 (reference)	0	0.14 (impurity)	1.0	1.0	1.0
2	1.38	0.14 "	1.0	1.06	1.06
3	2.94	0.14 "	1.04	1.08	1.13
4	0.1	0.83	0.91	0.86	0.91
5	0.1	0.4	0.71	0.97	0.93
6	0.1	0.91	1.04	0.91	1.02
7	1.24	0.41	1.08	1.13	1.18
8	0.35	0.38	1.04	1.22	1.13
9	0.56	0.54	1.12	1.22	1.17

Table II shows that the relative reaction rate was highest at high concentration of ammonia (20%) when both cobalt oxide and titanium oxide were added as promoters (compare sample 7-9 with sample 1).

Furthermore, the table II shows that by adding either cobalt oxide or titanium oxide alone, together with the other conventional promoters, a lower relative reaction rate at high concentration of ammonia was obtained (compare samples 2-3 and 4-6 with sample 1).

Furthermore, table II shows that the activity was increased at temperatures down to 350°C when both cobalt- and titanium oxide were added as extra promoters (see sample 7-9).

The example also shows that the activity was increased both at low and high reaction temperature when both cobalt- and titanium oxide were added as promoters.

CLAIMS:

1. A catalyst for the synthesis of ammonia from hydrogen and nitrogen consisting of iron oxides and promoters characterised in that the promoters comprise oxides of both cobalt and titanium in addition to Al, K, Ca and Mg oxides.
2. A catalyst for the synthesis of ammonia from hydrogen and nitrogen according to claim 1, characterised in that, the concentration of cobalt is between 0.1% and 3.0 % by weight of metal and the concentration of titanium is between 0.1% and 1.0% by weight of metal.

PCT

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International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : B01J 21/06, C01C 1/04		A1	(11) International Publication Number: WO 99/46038 (43) International Publication Date: 16 September 1999 (16.09.99)
(21) International Application Number: PCT/NO99/00008 (22) International Filing Date: 11 January 1999 (11.01.99) (30) Priority Data: 19981118 13 March 1998 (13.03.98) NO (71) Applicant (for all designated States except US): NORSK HYDRO ASA [NO/NO]; N-0240 Oslo (NO). (72) Inventors; and (75) Inventors/Applicants (for US only): FUGLERUD, Terje [NO/NO]; Snarveien 1, N-3925 Porsgrunn (NO). SKAUGSET, Per, Torbjørn [NO/NO]; Borgeåsen Terrasse 15, N-3911 Porsgrunn (NO). (74) Agent: JOHNSEN, Venche, Høines; Norsk Hydro asa, N-0240 Oslo (NO).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>	
(54) Title: CATALYST FOR THE SYNTHESIS OF AMMONIA FROM HYDROGEN AND NITROGEN			
(57) Abstract The present invention relates to a catalyst for the synthesis of ammonia from hydrogen and nitrogen consisting of iron oxides and promoters where the promoters comprise oxides of both cobalt and titanium in addition to Al, K, Ca and Mg oxides, and where the concentration of cobalt is between 0.1 % and 3.0 % by weight of metal and the concentration of titanium is between 0.1 % and 1.0 % by weight of metal.			

ATTACHMENT A

DECLARATION AND POWER OF ATTORNEY FOR U.S. PATENT APPLICATION

() Original () Supplemental () Substitute (X) PCT () Design

As a below named inventor, I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that I verily believe that I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Title: "Catalyst for the synthesis of ammonia from hydrogen and nitrogen"

of which is described and claimed in:

- () the attached specification, or
 () the specification in the application Serial No. _____ filed _____;
 and with amendments through _____ (if applicable), or
 (X) the specification in International Application No. PCT/NO99/00008, filed 11 January 1999, and as amended on _____ (if applicable).

I hereby state that I have reviewed and understand the content of the above-identified specification, including the claims, as amended by any amendment(s) referred to above.

I acknowledge my duty to disclose to the Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim priority benefits under Title 35, United States Code, §119 (and §172 if this application is for a Design) of any application(s) for patent or inventor's certificate listed below and have also identified below any application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

COUNTRY	APPLICATION NO.	DATE OF FILING	PRIORITY CLAIMED
NO	19981118	13 March 1998	YES

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose information material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application.

APPLICATION SERIAL NO.	U.S. FILING DATE	STATUS: PATENTED, PENDING, ABANDONED

And I hereby appoint John T. Miller, Reg. No. 21,120; Michael R. Davis, Reg. No. 25,134; Matthew M. Jacob, Reg. No. 25,154; Jeffrey Nolton, Reg. No. 25,408; Warren M. Cheek, Jr., Reg. No. 33,367; Nils E. Pedersen, Reg. No. 33,145 and Charles R. Watts, Reg. No. 33,142, who together constitute the firm of WENDEROTH, LIND & PONACK, L.L.P., attorneys to prosecute this application and to transact all business in the U.S. Patent and Trademark Office connected therewith.

I hereby authorize the U.S. attorneys named herein to accept and follow instructions from _____ as to any action to be taken in the U.S. Patent and Trademark Office regarding this application without direct communication between the U.S. attorneys and myself. In the event of a change in the persons from whom instructions may be taken, the U.S. attorneys named herein will be so notified by me.

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Full Name of Seventh Inventor	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
Residence & Citizenship	CITY	STATE OR COUNTRY	COUNTRY OF CITIZENSHIP
Post Office Address	ADDRESS	CITY	STATE OR COUNTRY ZIP CODE

I further declare that all statements made herein of my own knowledge are true, and that all statements on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

1st Inventor: Terje Fuglerud Date 31.08.2000
 2nd Inventor: Per Skaugset Date 31.08.2000
 3rd Inventor: Per Torbjørn SKAUGSET Date _____
 4th Inventor: _____ Date _____
 5th Inventor: _____ Date _____
 6th Inventor: _____ Date _____
 7th Inventor: _____ Date _____

The above application may be more particularly identified as follows:

U.S. Application Serial No. _____ Filing Date _____
 Applicant Reference Number _____ Atty Docket No. _____
 Title of Invention _____
